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IS 12038-1 (2007): Vitreous and porcelain enamels - Release of lead and cadmium from enamelled ware in contact with food, Part 1: Method of test [CHD 9: Ceramicware]

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भारतीय मानक

विट्रियस और पोर्सिलेन इनेमल — खाद्य सामग्री संपर्क में
इनेमलित आधानों से सीसा और कैडमियम विमोचित होना

भाग 1 परीक्षण पद्धति
(पहला पुनरीक्षण)

Indian Standard

VITREOUS AND PORCELAIN ENAMELS —
RELEASE OF LEAD AND CADMIUM FROM
ENAMELLED WARE IN CONTACT WITH FOOD

PART 1 METHOD OF TEST
(*First Revision*)

ICS 25.220.50; 67.250; 97.040.60

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NATIONAL FOREWORD

This Indian Standard (Part 1) (First Revision) which is identical with ISO 4531-1 : 1998 'Vitreous and porcelain enamels — Release of lead and cadmium from enamelled ware in contact with food — Part 1 : Method of test' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Ceramicware Sectional Committee and approval of the Chemical Division Council.

This standard was first published in 1987 as 'Permissible limits and test methods for toxic materials released from enamelware in contact with food'. During revision, the Committee felt that it would be more convenient to split the standard into two parts by adoption of ISO 4531-1 : 1998 and ISO 4531-2 : 1998 on dual number basis. Part 1 covered method of test and Part 2 covered permissible limits.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard which is to be substituted in its place is listed below along with its degree of equivalence for the edition indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 4531-2 : 1998 Vitreous and porcelain enamels — Release of lead and cadmium from enamelled ware in contact with food — Part 2 : Permissible limits	IS 12038 (Part 2) : 2007 Vitreous and porcelain enamels — Release of lead and cadmium from enamelled ware in contact with food: Part 2 Permissible limits (first revision)	Identical

The technical committee responsible for the preparation of this standard has reviewed the provisions of the following International Standards and has decided that they are acceptable for use in conjunction with this standard:

<i>International Standard</i>	<i>Title</i>
ISO 385-2 : 1984	Laboratory glassware — Burettes — Part 2 : Burettes for which no waiting time is specified
ISO 648 : 1977	Laboratory glassware — One-mark pipettes
ISO 1042 : 1998	Laboratory glassware — One-mark volumetric flasks
ISO 2723 : 1995	Vitreous and porcelain enamels for sheet steel — Production of specimens for testing
ISO 2724 : 1973	Vitreous and porcelain enamels for cast iron — Production of specimens for testing
ISO 3585 : 1998	Borosilicate glass 3.3 — Properties
ISO 3696 : 1987	Water for analytical use — Specification and test methods
ISO 4788 : 2005	Laboratory glassware — Graduated measuring cylinders

(Continued on third cover)

Introduction

The problem of lead and cadmium release from enamelled ware requires effective means of control to ensure the protection of the population against possible hazards arising from the use of improperly formulated, applied and fired enamels and/or decorations on the food contact surfaces of enamelled ware used for the preparation, serving and storage of foodstuffs.

NOTE Articles which are highly coloured or decorated on their food contact surfaces or which have a high surface area/volume ratio are more likely than other articles to release lead and/or cadmium.

As a secondary consideration, different requirements from country to country for the control of the release of toxic materials from the surfaces of enamelled ware present non-tarif barriers to international trade in these commodities. Accordingly, there is a need to establish internationally accepted methods of testing enamelled ware for the lead and cadmium release.

An expert panel convened by the World Health Organization (WHO) met in Geneva, in June 1976, and recommended the adoption of sampling methods, testing procedures and limits for the release of toxic materials from ceramic ware [6]. A further meeting was convened by the WHO in November 1979 [7].

The method of test specified in this part of ISO 4531 is based on those WHO recommendations, because it was the sense of the WHO meeting that the term "ceramic" includes ceramics, glass, vitreous enamels and glass ceramics. The description of the test method is largely in accordance with EN 1388-2 dealing with the determination of the release of lead and cadmium from silicate surfaces other than ceramic ware.

The amount of lead and/or cadmium determined by the method of test specified in this part of ISO 4531 will not be less than, and in the vast majority of cases will be greater than, the quantities released into acidic foods and drinks over a period of time [8]. If WHO recommendations were to include hot testing at any time then a new edition of this part of ISO 4531 should be considered.

Indian Standard

**VITREOUS AND PORCELAIN ENAMELS —
RELEASE OF LEAD AND CADMIUM FROM
ENAMELLED WARE IN CONTACT WITH FOOD**

PART 1 METHOD OF TEST

(*First Revision*)

1 Scope

This part of ISO 4531 specifies a simulating method of test for determination of the release of lead and cadmium from enamelled ware which are intended to come into contact with food (including drinks).

This part of ISO 4531 is applicable to enamelled ware including tanks and vessels which are intended to be used for the preparation, serving and storage of food.

This part of ISO 4531 is applicable to enamelled ware including tanks and vessels which can be used for the preparation, serving and storage of food.

This part of ISO 4531 also specifies a method of test for determining the release of lead and/or cadmium from a drinking rim.

It is not applicable to ceramic ware, glassware or glass ceramic ware.

2 Normative References

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 4531. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 4531 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 385-2, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified*.

ISO 648, *Laboratory glassware — One-mark pipettes*.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.

ISO 2723, *Vitreous and porcelain enamels for sheet steel — Production of specimens for testing*.

ISO 2724, *Vitreous and porcelain enamels for cast iron — Production of specimens for testing*.

ISO 3585, *Borosilicate glass 3.3 — Properties*.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*.

ISO 4531-2, *Vitreous and porcelain enamels — Release of lead and cadmium from enamelled ware in contact with food — Part 2: Permissible limits*.

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*.

3 Definitions

For the purposes of this part of ISO 4531, the following definitions apply.

3.1

vitreous enamel (USA: porcelain enamel)

substance resulting from smelting or fritting of inorganic compounds to form a vitreous material fused, or capable of being fused (see frit), in one or more layers on to a metallic base

3.2

enamelled ware

metallic articles coated with vitreous and porcelain enamel

3.3

foodware

articles which are intended to be used for the preparation, cooking, serving and storage of food or drinks including tanks and vessels

3.4

flat ware

articles which cannot be filled and articles which can be filled, the internal depth of which, measured from the lowest point to the horizontal plane passing through the upper rim, does not exceed 25 mm

NOTE Flatware also includes the test specimens for testing vitreous porcelain **enamelled equipment, containers and water heaters**.

3.5

hollow ware

articles which can be filled the internal depth of which, measured from the horizontal plane passing through the lowest point of the upper rim, exceeds 25 mm, excepting hollow ware items having a filling volume exceeding 3 litres which are classed as storage vessels; examples of such articles are kitchen utensils such as pots, pans and kettles

3.6

cooking ware

foodware, specifically intended to be heated in the course of preparation of food and drinks by means such as steaming, boiling, braising, stewing, roasting, baking or by microwaves; examples of such articles are for instance casseroles, bakers, roasters, soufflés, percolators and saucepans

3.7

storage vessel

tank

articles with a capacity of equal to or greater than 3 litres

3.8

drinking rim

20 mm wide section of the external surface of a drinking vessel, measured downwards from the upper edge along the wall of the vessel

3.9

test solution

solution used to extract lead and cadmium from silicate surfaces

3.10

extract solution

aqueous acidic solution obtained from the exposure of a silicate surface to the test solution

3.11

sample measuring solution

solution used for measuring the concentration of the analyte, and perhaps the extract solution or an appropriately diluted extract solution

3.12

analyte

element or constituent to be determined

3.13

stock solution

solution of appropriate composition containing the analyte, in a known high concentration

3.14

standard solution

solution containing the analyte, in known concentration suitable for the preparation of calibration solutions

3.15

set of calibration solutions

set of simple or synthetic calibration solutions having different analyte concentrations. The zero number is, in principle, the solution having zero concentration of the analyte

3.16

atomic absorption spectrometry (AAS)

method for determining chemical elements based on measurement of the absorption of characteristic electromagnetic radiation by atoms in the vapour phase

3.17

optimum working range

range of concentrations of an analyte in solution over which the relationship between absorption and concentration is linear, or sufficiently approximates to linearity so that any divergence at the prescribed limit of concentration does not discernably affect any analytical determination

3.18

direct method of determination

analytical-curve technique

method consisting of inserting the measure obtained in the analytical function, and deducing from it the concentration of the analyte

3.19

analytical function

calibration function

function relating the value of the concentration to the characteristic value obtained from the set of calibration solutions

NOTE The graph of this function is called the "analytical curve (calibration graph)"

3.20

bracketing technique

method consisting of bracketing the measured absorbance or intensity of the sample solution between two measurements made on calibration solutions of neighbouring concentrations

4 Principle

Enamelled surfaces are placed in contact with 4 % (V/V) acetic solution for 24 h at 22 °C to extract lead and/or cadmium, if present, from the surfaces of the articles or test specimens.

The proportions of extracted lead and cadmium are determined by flame atomic absorption spectrometry (FAAS).

NOTE In routine tests, other equivalent analysis methods can be used.

5 Reagents

During the determination, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696).

It is permissible to prepare proportionately greater quantities of test solution and analytical solutions than specified in 5.2, 5.4 and 5.5.

5.1 Acetic acid, (CH_3COOH) , glacial, density $\rho = 1,05 \text{ g/ml}$.

5.2 Test solution, acetic acid, 4 % (V/V) solution.

By means of a graduated measuring cylinder (6.7) add, to 500 ml of water, 40 ml $\pm 1 \text{ ml}$ of glacial acetic acid (5.1) and make up to 1 l. Prepare the test solution freshly prior to use and in sufficient quantity to enable the whole of any group of tests and analysis to be completed.

5.3 Analytical stock solutions

5.3.1 Stock lead solution (1 g of lead per litre)

Prepare an analytical stock solution containing $1\,000 \text{ mg} \pm 1 \text{ mg}$ of lead per litre in the test solution (5.2).

Alternatively, appropriate commercially available lead solutions may be used, provided that the concentrations of such solutions are known to an equivalent accuracy.

1 ml of this stock solution \equiv mg of lead.

5.3.2 Stock cadmium solution (1 g of cadmium per litre)

Prepare an analytical stock solution containing $1\,000 \text{ mg} \pm 1 \text{ mg}$ of cadmium per litre in the test solution (5.2).

Alternatively, appropriate commercially available lead solutions may be used, provided that the concentrations of such solutions are known to an equivalent accuracy.

1 ml of this stock solution \equiv 1 mg of cadmium.

5.4 Analytical standard solutions

5.4.1 Standard lead solution (0,1 g of lead per litre)

By means of a one-mark pipette (6.4), transfer 10 ml of the stock lead solution (5.3.1) to a 100 ml one-mark volumetric flask (6.3), make up to the mark with the test solution (5.2) and mix well. Renew this solution every four weeks.

1 ml of this standard solution \equiv 0,1 mg of lead.

From this solution prepare appropriate calibration solutions by dilution with the test solution (5.2) using the burette (6.5), and keep them in suitably prepared containers. Renew these solutions every four weeks. It is also permissible to prepare lead calibration solutions directly from the stock solution by using one-mark glass pipettes or precision piston pipettes with a fixed stroke and 500 ml to 2 000 ml volumetric flasks.

5.4.2 Standard cadmium solution (0,01 g of cadmium per litre)

By means of a one-mark pipette (6.4), transfer 1 ml of the stock cadmium solution (5.3.2) to a 100 ml one-mark volumetric flask (6.3), make up to the mark with the test solution (5.2) and mix well. Renew this solution every four weeks.

1 ml of this standard solution = 0,01 mg of cadmium.

From this solution prepare appropriate calibration solutions by dilution with the test solution (5.2), using the burette (6.5), and keep them in suitably prepared containers. Renew these solutions every four weeks. It is also permissible to prepare cadmium solutions directly from the stock solution by using one-mark glass pipettes or precision piston pipettes with a fixed stroke and 500 ml to 2 000 ml volumetric flasks.

5.5 Paraffin wax, high melting point.

NOTE Suitable wax is specified e. g. in the European Pharmacopoeia.

5.6 Washing agent, commercially available non-acidic manual dishwashing detergent in common dilution.

5.7 Silicone sealant, in a tube or dispenser, enabling a ribbon of silicone sealant approximately 6 mm to be formed.

6 Apparatus

Use only laboratory glassware, complying with the requirements of appropriate International Standards, where they exist, and made of borosilicate glass as specified in ISO 3585.

6.1 Flame atomic absorption spectrometer, with a detection limit of Pb equal to or smaller than 0,1 mg/l (in 4 % (V/V) acetic acid) and of Cd equal to or smaller than 0,01 mg/l (in 4 % (V/V) acetic acid) where the detection limit is the mass concentration of analyte for which the absorbance is three times the standard deviation of the background noise of the system.

NOTE The background noise of the system can be derived either from a series of absorbance measurements made on a solution which contains lead or cadmium at a concentration distinctly detectable above, but close to, the composition of solvent blanks, or directly by suitable flame atomic absorption spectrometers measuring the absorbance of a solvent blank.

6.2 Line sources, for lead and cadmium.

6.3 One-mark volumetric flasks, capacities 100 ml and 1 000 ml, complying with the requirements specified for class B or better one-mark volumetric flasks in ISO 1042. Other sizes of one-mark volumetric flasks may also be required.

6.4 One-mark pipettes, capacities 10 ml and 100 ml, complying with the requirements specified for class B or better one-mark pipettes in ISO 648. Other sizes of one-mark pipettes may also be required.

6.5 Burette, capacity 25 ml, graduated in divisions of 0,05 ml, complying with the requirements specified for class B or better burettes in ISO 385-2.

6.6 Covers, for the articles under test, e. g. plates, watch-glasses, petri dishes of different sizes and opaque if no darkroom is available.

6.7 Graduated measuring cylinders, capacities 50 ml and 500 ml, complying with the requirements specified in ISO 4788. Other sizes of graduated measuring cylinders may also be required.

6.8 Straightedge, strip of metal, or other material, having at least one clearly identifiable edge, cut straight and which does not deviate from straightness by the equivalent of more than one millimetre in one metre. The straight edge may incorporate a spirit level.

6.9 Depth-gauge or rule, calibrated in millimetres, to be used in conjunction with the straight edge (6.8).

7 Samples

The laboratory sample shall consist of four similar single articles, identical in material, shape, dimensions and decoration.

For testing vitreous and porcelain enamelled tanks and vessels, test specimens prepared in accordance with ISO 2723 or ISO 2724 shall be used. These test specimens shall be tested as flat ware.

8 Preparation of samples

8.1 Distinction between flat ware and hollow ware

From a group of identical articles, select a single sample and place it on a flat horizontal surface. Place the straight edge (6.8) on the rim of the article. Where the article has a spout, place the straight edge horizontally from the lowest point of the spout. Measure the distance, h , between the lowest point in the interior of the article and the straight edge. Articles with a value of h not exceeding 25 mm are deemed to be flat ware, whilst articles with a value of h exceeding 25 mm are deemed to be hollow ware. If an article is a borderline case between flat ware and hollow ware, it shall be classified as flat ware.

8.2 Determination of reference surface area for flat ware

Invert the sample on graph paper marked in millimetre squares and draw the contour round the rim. Calculate or measure the area enclosed by the contour and record this as the reference area S_R in square decimetres to two decimal places. For circular articles the reference surface area shall be calculated from the diameter of the article. Determine the reference area for non-fillable ware accordingly, measuring the uncovered surface to be extracted (8.4 a) or 8.4 b) only.

NOTE This method of measurement compensates for the effect of curvature of the articles.

8.3 Cleaning of the samples

Wash the samples briefly in an aqueous solution at $40^{\circ}\text{C} \pm 5^{\circ}\text{C}$ containing 1 ml/l of washing agent (5.6). Rinse the samples thoroughly with tap water, then with distilled water and allow to drain. Wipe dry with clean filter paper. Do not use any sample which shows residual staining.

If an area of the surface of the sample is not intended to come into contact with foodstuffs in normal use, other than the interior of any lid (see 8.4 a) and 8.4 b)), cover this area after the initial washing and drying with a protective coating withstanding the effect of the test solution (5.2) and not releasing any detectable levels of lead and cadmium into the test solution during the test procedure.

Do not handle the surface to be tested after it has been cleaned or prepared.

8.4 Preparation of articles which cannot be filled

Articles which cannot be filled to 6 mm from overflowing as specified in 9.2.2.1 shall be regarded as non-fillable.

- a) Coat these articles carefully on all surfaces except the reference surface (see 8.2) with melted paraffin wax (5.5) and test as specified in 9.2.2.2 a) or
- b) Apply a ribbon of silicone sealant (5.7), 6 mm wide, to the enamelled surface of the article around the whole perimeter. Inspect the ribbon and ensure that the ribbon of sealant is complete and is in intimate contact with the enamelled surface around the whole perimeter. Check that the height of the sealant ribbon is approximately 4 mm and is sufficient to allow an adequate bulk of test solution (5.2) to be added. Set the article aside overnight in order for the silicone sealant to cure and test as specified in 9.2.2.2 b).

8.5 Drinking rim

Determine the region of the drinking rim by inverting the article on a smooth horizontal surface and rotating it against a felt tipped pen fixed at a height corresponding to the boundary of the drinking rim (3.8).

Cover carefully the portion of the external surface of the article that is not to be tested with melted paraffin wax (5.5). The cover and handle present in the drinking rim region shall be tested in the same way. It is permissible for the drinking rim to be cut off and tested separately.

9 Procedure

9.1 General

When lead alone shall be determined, carry out the extraction under normal laboratory lighting conditions. When cadmium shall be determined, alone or together with lead, carry out the extraction in the dark.

9.2 Extraction

9.2.1 Test temperature

Condition the sample or test specimen and the test solution (5.2) to $22^{\circ}\text{C} \pm 2^{\circ}\text{C}$, prior to 9.2.2.

9.2.2 Contact between the samples and the test solution

9.2.2.1 Fillable samples

Place the sample on a flat, horizontal surface and fill it, by means of a graduated measuring cylinder (6.7), with the test solution (5.2) to a level, not more than 1 mm below the overflow point. In the case of samples having a flat or sloping rim, ensure that the distance between the surface of the liquid and the overflow point does not exceed 6 mm, measured along the sloping rim. In case of flat ware, determine the volume V of the test solution to an accuracy of 2 % after the extraction (i. e. filling volume). Cover and keep in the dark if cadmium shall be determined.

9.2.2.2 Non-fillable samples

a) When prepared according to 8.4 a):

- Place the samples in a suitable vessel, e. g. a borosilicate glass vessel of suitable size and add by means of a graduated measuring cylinder (6.7) the test solution (5.2) to cover the samples completely.
- Record the required volume V of test solution (contact volume) to an accuracy of 2 %. Cover the vessel with a suitable cover (6.6) and, if cadmium shall be determined, place it in the dark.

b) When prepared according to 8.4 b):

- Place the article, with sealant, on a flat, horizontal surface and add the test solution (5.2) by means of a graduated measuring cylinder (6.7).
- Record the volume V of test solution used (contact volume) to an accuracy of 2 %. Cover the article and test solution with a suitable cover (6.6) and, if cadmium shall be determined, place it in the dark.

NOTE Standard articles can be exposed to the test solution (5.2) when fitted into a silicone rubber mould in which the articles fit forming a watertight seal and so constructed as to encroach 6 mm from the rim of the article and forming a depth no greater than 7 mm.

9.2.2.3 Lids of receptacles

If an article is provided with a lid, the inner surface of the lid shall be also tested, on its own.

9.2.2.4 Drinking rim

Place the prepared sample (see 8.5) with the rim downwards, in a flat-bottomed vessel. Cover the rim completely with the test solution (5.2).

If the rim is cut off, place the separated rim in a flat-bottomed vessel not less than 25 mm in height (e. g. petri dish or beaker) and cover completely with the test solution.

Ensure that the size of the vessel and the height of the covering test solution are such that the volume of test solution is kept to a minimum; determine the volume of test solution used to an accuracy of 2 %.

9.2.3 Duration of extraction

Maintain the filled, or immersed, samples at $22^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for $24\text{ h} \pm 30\text{ min}$ under conditions which preclude evaporative losses.

NOTE Plastic trays with close fitting lids are suitable containers to prevent evaporative losses from the test vessels. Particular care should be taken to ensure that evaporative losses of test solution (5.2) are minimized. If necessary, such losses can be checked for by weighing.

9.3 Sampling the extract solution for analysis (sample measuring solution)

Homogenize the extract solution, by stirring or other methods, without loss of solution or causing abrasion of the surface to be tested.

NOTE A suitable method for homogenizing the extract solution consists in removing a quantity by pipette and allowing it to run back into, or on to, the sample several times, avoiding dilution or evaporation loss in the process.

Transfer the extract solution or a part of it to a suitable vessel. This is the sample measuring solution. Perform the analysis as quickly as possible.

9.4 Calibration and determination

9.4.1 Calibration

Set up the flame atomic absorption spectrometer having regard to the manufacturer's instructions using wavelengths of 217,0 nm for lead determination, and 228,8 nm for cadmium determination with appropriate correction for background absorption effects.

NOTE Where appropriate, a wavelength of 283,3 nm can be used for the analytical confirmation of lead.

Aspirate the zero member of the set of the calibration solutions (i. e. the test solution (5.2) and adjust zero. Aspirate the set of calibration solutions, prepared by dilution of the standard solutions (see 5.4) with the test solution (5.2).

Suggested ranges:

- 0,2 mg/l to 10,0 mg/l Pb
- 0,02 mg/l to 0,5 mg/l Cd

Aspirate test solutions (5.2) after each calibration solution and record the absorbance values obtained.

9.4.2 Determination

Set up the spectrometer as specified above. Aspirate distilled water and then test solution (5.2) and make sure that the absorbance is zero. Aspirate the sample measuring solutions (see 9.3), interspersed with test solution (5.2). Measure the absorbance values of the sample measuring solutions.

If the mass concentration of lead in the sample measuring solution exceeds 10 mg/l or if the mass concentration of cadmium exceeds 0,5 mg/l, dilute a suitable aliquot portion of the extract solution with test solution to give a mass concentration of lead less than 10 mg/l, and a mass concentration of cadmium of less than 0,5 mg/l, so that the signals are in the optimum working range. Measure each sample measuring solution at least three times, and use the mean value for calculation.

To increase accuracy, especially when repeated measurement of a calibration solution with a mean metal concentration indicates minimal drift, employ the bracketing technique (3.20). For this purpose, following a preliminary test yielding approximate values for the mass concentrations of lead and/or cadmium in the calibration solutions, use calibration solutions in which the metal ions are present in mass concentrations as closely as possible above and below those of the sample measuring solution.

10 Expression of results

10.1 Bracketing technique

Calculate the lead or cadmium concentration, C_0 , expressed in milligrams per litre of the extract solution by equation (1):

$$C_0 = \left[\left(\frac{A_0 - A_1}{A_2 - A_1} \right) (C_2 - C_1) + C_1 \right] d \quad \dots (1)$$

where

A_0 is the absorbance corresponding to lead or cadmium in the sample measuring solution;

A_1 is the absorbance corresponding to lead or cadmium in the lower bracketing solution;

A_2 is the absorbance corresponding to lead or cadmium in the upper bracketing solution;

C_1 is the lead or cadmium concentration of the lower bracketing solution, in milligrams per litre;

C_2 is the lead or cadmium concentration of the upper bracketing solution, in milligrams per litre;

d is the factor, used if the sample extract solution was diluted.

10.2 Calibration curve technique

Read the lead or cadmium concentration, expressed in milligrams per litre of the sample measuring solution, directly from the calibration curve.

10.3 Calculation of release of lead and cadmium for flat ware, non-fillable articles and test specimens

Calculate the lead or cadmium released per unit surface area from flat ware and non-fillable articles or test specimens, R_{Pb} or R_{Cd} , expressed in milligrams per square decimetre and by the equations (2) and (3), respectively:

where

$$R_{\text{Pb}} = \frac{C_0 \cdot V}{S_R} \quad \dots (2)$$

$$R_{\text{Cd}} = \frac{C_0 \cdot V}{S_R} \quad \dots (3)$$

C_0 is the mass concentration of lead and cadmium of the extract solution, calculated as specified in 10.1 or 10.2, in milligrams per litre;

V is the volume of test solution used for the extraction (see 9.2.2 and 9.2.3 respectively), in litres;

S_R is the reference surface area of the article (see 8.2), in square decimetres.

10.4 Calculation of release of lead and cadmium from the drinking rim

Calculate the release of lead or cadmium from the drinking rim of an article, $A_{0,T}$ in milligrams per article by equation (4):

$$A_{0,T} = C_0 \cdot V \quad \dots(4)$$

where

C_0 is the mass concentration of lead and cadmium of the extract solution, calculated as specified in 9.2.2.4, in milligrams per litre;

V is the volume of test solution used for the extraction, in litres.

10.5 Calculation of release of lead and cadmium for receptacles with lids

Calculate the release of lead or cadmium from both container and total inner surface of the lid, separately, in milligrams per container and per lid, respectively. The values shall be added and the sum shall be related, depending on the case concerned, to the surface area, expressed in milligrams per square decimetre, or to the volume of the container alone, expressed in milligrams per litre.

10.6 Reporting

For hollow ware, report the results to the nearest 0,1 mg/l for lead, and to the nearest 0,01 mg/l for cadmium.

For flat ware (and non-fillable articles and test specimens), report the results to the nearest 0,1 mg/dm² for lead and to the nearest 0,01 mg/dm² for cadmium.

For a drinking rim, report the release to the nearest 0,1 mg per rim for lead, and to the nearest 0,01 mg per rim for cadmium.

If a receptacle with lid is tested, report the sum of the two extract values to the nearest 0,1 mg/l and 0,1 mg/dm² respectively for lead and to the nearest 0,01 mg/l and 0,01 mg/dm² respectively for cadmium.

10.7 Comparison with permissible limits

An article is deemed to comply with specified permissible limits, when these are not exceeded by the values of lead and/or cadmium released, determined as specified in this part of ISO 4531.

For receptacles with lids the permissible limit for the lead and/or cadmium released, in milligrams per square decimetre or in milligrams per litre, is deemed to be the value applicable to the receptacle alone.

If the results from an article exceed specified permissible limits, but by no more than 50 %, the article concerned is nevertheless deemed to comply with the permissible limits if at least three other similar articles tested under the conditions specified in this part of ISO 4531 provide results such that the arithmetic mean of lead, or of cadmium, released from these articles does not exceed the permissible limit or limits and none of the articles exceeds the permissible limit or limits by more than 50 %.

11 Test report

The test report shall contain the following information:

- a) reference to this part of ISO 4531, i.e. ISO 4531-1;
- b) an identification of the articles tested, including their type, origin and designation;
- c) the place and date of sampling;
- d) the date of receipt and date of testing the sample;
- e) the surface area and the reference surface area, and the filling volume or contact volume for non-fillable articles;
- f) the number of samples tested;
- g) each single result, in accordance with 10.6, and the mean value for each article or test specimen, expressed as milligrams of lead and/or cadmium per square decimetre of surface area for flat ware, or milligrams of lead and/or cadmium per litre of volume for hollow ware;
- h) for a receptacle with a lid, the sum of the two extract values, i. e. for the receptacle and for lid tested separately;
 - i) for a drinking rim, the release of lead and/or cadmium in milligrams per article rim;
 - j) any operation not included in this part of ISO 4531, or regarded as optional;
 - k) whether the article tested satisfies the requirements for permissible limits of release as specified in ISO 4531-2.

NOTE Any unusual features noted during the determination should also be reported.

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- [1] ISO 385-1:1984, *Laboratory glassware – Burettes – Part 1: General requirements*.
- [2] ISO 6486-1:1986, *Ceramic ware, glass-ceramic ware and glass flatware in contact with food – Release of lead and cadmium – Part 1: Method of test*.
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- [4] ISO 7086-1:1982, *Glass hollow ware in contact with food – Release of lead and cadmium – Part 1: Test method*.
- [5] EN 1388-2:1995, *Materials and articles in contact with foodstuffs - Silicate surfaces - Part 2: Determination of the release of lead and cadmium from silicate surfaces other than ceramic ware*.
- [6] WHO/Food Additives 77.44. *Ceramic Foodware Safety, Sampling, Analysis and Limits for Release* (Report of a WHO meeting, Geneva, 8 – 10 June 1976).
- [7] WHO/Food Additives HCS/79.7. *Ceramic Foodware Safety, Critical Review of Sampling, Analysis and Limits for Lead and Cadmium Release* (Report of a WHO meeting, Geneva, 12 – 14 November 1979).
- [8] FREY, E., and SCHOLZE, H., *Blei- und Cadmiumlässigkeit von Schmelzfarben, Glasuren und Emails in Kontakt mit Essigsäure und Lebensmitteln und unter Lichteinwirkung* [Lead and cadmium release from fused colours, glazes and enamels in contact with acetic acid and foodstuffs and under the influence of light]. Bericht Deutsche Keramische Gesellschaft, 1979 (vol. 56), pp. 293-297.

(Continued from second cover)

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounding off value should be the same as that of the specified value in this standard.

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